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Enhancing Hydrophilicity and Efficiency of PVC-Based Nanofiber Membranes by Adding PEG, Chitosan, and Silver Nanoparticles for Water Filtration †

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Abstract: This study aims to investigate PVC-based nanofiber membranes added with PEG, chitosan, and silver nanoparticles (CSNPs and AgNPs) to improve their hydrophilicity and filtration efficiency. The nanofiber membranes were fabricated by an electrospinning technique. All nanofiber membranes were subjected to a tensile test according to the ASTM D882 standard and water contact angle (WCA) measurement. SEM was used to examine nanofiber morphology. Adding PEG to PVC increased hydrophilicity, lowering the water contact angle from 135° to 83°, while adding CSNPs and AgNPs to PEG/PVC slightly decreased it. The inclusion of these nanoparticles improved membrane tensile strength. In filtration tests, 0.5% AgNPs/PEG/PVC worked better against Colitinja bacteria than the CSNP variant. However, both types of nanoparticles were equally effective at inhibiting *E. coli*. These results indicate that 0.5% CSNP/PEG/PVC and 0.5% AgNPs/PEG/PVC membranes are promising for antibacterial water filtration applications due to their hydrophilic and durable properties.

Keywords: PVC; PEG; CSNPs; AgNPs; electrospinning; nanofiber membrane; water contact angle; water filtration

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1. Introduction

Clean water is a fundamental human need; however, water pollution resulting from industrial development poses a severe health risk, particularly in rural areas [1,2]. In relation to water pollution, urgent research is required for the development water filtration technology. In this case, nanotechnology offers nanofiber membranes made from conductive polymer solutions using the electrospinning method. The resulting membrane has a nanometer-scale fiber structure (200–500 nm) and a high sub-micrometer-scale porosity density, making it very effective for filter materials.

Tang et al. [3] have reviewed the manufacture of nanometer-scale fibers using various polymer solutions and methods, namely, electrospinning, dry spinning, wet spinning, emulsion spinning, melt spinning, and phase-separation spinning. Among these methods, electrospinning stands out for its high productivity, ease of operation, high porosity density in the resulting membrane, and interconnection of fibers to form a network known as crosslinked fibers.

Air and water filter media can use nanofiber membranes, but for water filter applications, the membrane must be hydrophobic. There have been some studies on various nanofiber membranes, including polyvinyl chloride (PVC)-based membranes that are used in water filters [3,4]. PVC has a wide range of applications, one of which is water filtration. However, the natural properties of the chloride functional group make PVC super hydrophobic [5], causing pure PVC to not be directly used for water filtration. It needs to be combined with hydrophilic polymers, including polyvinylpyrrolidone (PVP) and polyvinyl oxide (PEO). Previous studies have reported PVP/PVC and PEO/PVC nanofiber membranes for water filter applications [6–8]. Furthermore, the hydrophilic, flexible, and non-toxic properties of polyethylene glycol (PEG) make it a popular choice for biomaterial applications. Adding Ag nanoparticles (AgNPs) to PEG/cellulose acetate (CA) nanofiber membranes has helped to determine their mechanical and physical properties [9], but this research has not been used on water filters. The tensile test results showed that adding PEG to CA and then Ag nanoparticles to PEG/CA increased the tensile stress (from 0.34 MPa to 0.04 MPa) and tensile modulus (from 0.48 MPa to 0.95 MPa). A similar pattern happened when PEO was added to PVC and then chitosan nanoparticles (CSNPs) were added to PEO/PVC. In this case, the tensile strength of the PEO/PVC membrane (1 MPa) [8] was higher than that of the PEG/CA membranes. However, in terms of the membrane's ability to eliminate bacteria, the membrane containing AgNPs can eliminate *E. coli* and *S. aureus* bacteria by 100% within one hour. This study's information has inspired researchers to take advantage of PEG's opportunities.

On the other hand, nanofiber membranes that are made from 15% PVC dissolved in DMAC have a contact angle of 135° [6,8] and are very good at keeping water away. The addition of PVP reduces the contact angle to 129.81° at 2 wt.% and 123.25° at 5 wt.% [6], indicating a shift towards hydrophilic properties. However, according to Asmatulu et al. [7], the addition of 5 wt.% PVP to PVC showed a much lower contact angle (16°), so it was much more hydrophilic. Meanwhile, the addition of PEO to PVC [8] showed a decrease in the contact angle with a value slightly lower than the results of Alarifi et al. [6], which is ~96° for the addition of 4 wt.% PEO.

Additionally, PEG is considered compatible with PVC due to its non-toxic properties, flexibility, and frequent use in biomaterial applications [9]. PEG is also used to enhance the hydrophilicity of poly-caprolactone (PCL) [10–12]. Furthermore, PEG serves as a stabilizing agent [13]. Majumder et al.'s [9] research reveals that electrospinning can process PEG with a molecular weight of 200 g/mol because electrospinning PEG with a molecular weight of 600 g/mol was difficult. Previous studies have shown that PEG is a functional hydrophilic polymer, and PEG/PVC blends hold significant research potential. However, there are no reports of nanofiber membranes made of a mixture of PEG and PVC for water filtration applications. Therefore, this study characterized the mechanical and physical properties of PVC, PEG/PVC, chitosan nanoparticles (CSNPs)/PEG/PVC, and AgNPs/PEG/PVC nanofiber membranes, and used them for water filtration. This study examined the hydrophilicity of membranes, their capability to inhibit bacteria, and the efficiency of water filtration. This work also studied the relationship between nanofiber morphologies and their tensile strength to understand the mechanical strength of the PEG/PVC-based membranes for water filter applications.

2. Materials and Methods

PVC (high molecular weight/Mw), PEG (Mw: 5000 g/mol), and AgNPs were purchased from Sigma Aldrich (USA), while N, N-Dimethylacetamide (DMAC), and chitosan nanoparticles (CSNPs) (~50 nm) were supplied from EMSURE, Germany, and ANHUI MINMETALS DEVELOPMENT I/E Co., Ltd., China, respectively. The particle-sized CSNPs were confirmed by transmission electron microscopy (TEM) [14] and CSNPs are present in their semicrystalline phase.

This study prepared four different types of polymer solutions: (1) PVC (15%) using N-Dimethylacetamide (DMAC) as a solvent; (2) PEG/PVC with PEG concentrations of 1, 2, and 3%; (3) CSNPs/PEG/PVC; and (4) Ag nanoparticles (AgNPs)/PEG/PVC, adding 0.5, 1.0, and 1.5% of CSNPs and AgNPs, respectively, to the best PEG/PVC solution. Polymer solutions of PVC and PEG/PVC were prepared according to the ratios depicted in Table 1.

Table 1. The preparation of PVC and PEG/PVC polymer solutions.

No.	Polymer Solution	Ratio (*w*/*w*)
1	PVC/DMAC/PEG	15%:85%:0%
2	PVC/DMAC/PEG	14%:85%:1%
3	PVC/DMAC/PEG	13%:85%:2%
4	PVC/DMAC/PEG	12%:85%:3%

PVC solution was prepared by dissolving it in DMAC at a ratio of 15:85% (*w*/*w*), mixing them on a hot plate stirrer at 400 rpm and 60 °C for an hour, and then cooling to room temperature. The PEG/PVC solution was prepared by mixing PEG into the PVC solution at room temperature for 60 min using a magnetic stirrer. Each of the CSNPs and AgNPs was added to the PEG/PVC solution and mixed with a magnetic stirrer for 30 min to create a homogeneous suspension. All polymer solutions and suspensions were then fabricated into nanofiber membranes using the electrospinning method, operating at optimized parameters, i.e., a voltage ranging from 11 to 15 kV, a needle diameter of 8 mm, and a distance from a needle tip to a collector plate (TCD) ranging from 12 to 14 cm.

All nanofiber membranes were then subjected to water contact angle measurements and a tensile test, following the ASTM D882 standard, using a universal testing machine (UTM, Zwick 0.5). Five to eight membrane specimens were prepared for each test parameter. In addition, we used a scanning electron microscope (SEM, JSM-6510LA) to observe the morphology of nanofiber membranes and Ag nanoparticles. The average nanofiber diameter was measured at 100 nanofibers for each membrane specimen. Optimization was subsequently carried out on all measured and tested membranes. The selected membranes containing CSNPs and AgNPs were used for the water filtration test. In this study, the groundwater used for water filtration was obtained from Godean, Yogyakarta, Indonesia. The well is located on the cattle pen's side (Figure 1a). The Health and Calibration Laboratory Center, Yogyakarta Health Service, Indonesia, conducted the test for the groundwater containing bacteria (*E. coli* and Colitinja) both before and after filtration. The filtration process (Figure 1b) without pressure was conducted for approximately 9 h to obtain 100 mL of filtered water for the water test. A water test both before and after filtration was conducted and the used nanofiber membranes were examined by SEM. Additionally, the water filtration efficiency was calculated using the following equation:

$$\eta = [(N_{before} - N_{after})/N_{before}] \times 100\% \quad (1)$$

where η is the water filtration efficiency and N_{before} and N_{after} are the number of bacteria before and after filtration (MPN), respectively. MPN is a unit of the most probable number.

(**a**) (**b**)

Figure 1. (**a**) Groundwater intake location for filtration testing and (**b**) water filtration process.

3. Results and Discussion

3.1. Water Contact Angle

Table 2 shows the types of nanofiber membranes used, as well as the measurement and testing results (contact angle, nanofiber diameter, and tensile properties). The results of the water contact angle (Table 2 and Figure 2) indicated that adding PEG decreases the contact angle, meaning that it changes the hydrophobic properties to become more hydrophilic. The addition of 2% PEG showed a significant decrease in the contact angle. In this experiment, adding a PEG concentration higher than 2% increased the viscosity of the PEG/PVC solution, making it unable to be electrospun. Therefore, antibacterial nanoparticles (CSNPs and AgNPs) were only added to 2PEG/PVC. However, the addition of CSNPs and AgNPs increased the contact angle by 4.5–7.4%. These values are still lower than the previous studies, namely the addition of 1% CSNPs to 4PEO/PVC (94.18°) [8], suggesting that the addition of PEG to PVC is more effective as a water filter membrane than PEO/PVC membranes. However, the addition of 4% polyvinylpyrrolidone (PVP) to PVC (4PVP/PVC)[7] showed a much lower water contact angle (58.51°) than 2PEG/PVC and 4PEO/PVC. Additionally, Alarifi et al. [6] showed a water contact angle of 123° for the same material as Asmatulu et al. [7].

Table 2. The measurement and testing results of the nanofiber membranes.

Nanofiber Membranes	Water Contact Angle (degree)	Average Nanofiber Diameter (nm)	Tensile Properties		
			Tensile Strength (MPa)	Young Modulus (MPa)	Tensile Strain (%)
PVC	133	196	2.04	32.61	25.06
1PEG/PVC	110	-	-	-	-
2PEG/PVC	83	214	3.22	34.64	21.42
0.5CSNP/2PEG/PVC	86.95	223	3.65	37.85	33.71
0.5AgNP/2PEG/PVC	89.60	235	3.38	43.42	42.60

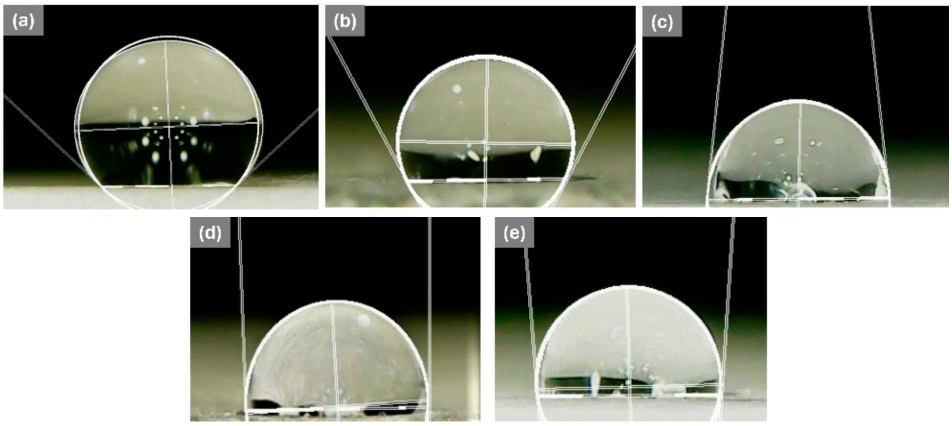

Figure 2. The water contact angle of all membrane nanofibers. (**a**) PVC, (**b**) 1PEG/PVC, (**c**) 2PEG/PVC, (**d**) 0.5CSNP/2PEG/PVC, and (**e**) 0.5AgNP/2PEG/PVC.

3.2. Characterization of Nanofiber Morphology and Tensile Properties

Based on the results, the morphology of the four membranes (PVC, 2PEG/PVC, 0.5CSNP/2PEG/PVC, 0.5AgNP/2PEG/PVC) (Figure 3) needs to be characterized to determine their effects on the tensile properties (Figure 4). The average nanofiber diameter (Table 2) increased by adding PEG, CSNPs, and AgNPs, as seen in the SEM images (Figure 3). The nanofibers formed in all the membranes are mostly continuous, with only a few slightly curved fibers. The average nanofiber diameter of 2PEG/PVC and 0.5CSNP/2PEG/PVC (214 nm and 223 nm) was smaller than 4PEO/PVC (357 nm) and 1CSNPs/4PEO/PVC (257 nm) [8]. Therefore, the tensile strength of the former was higher than that of the latter. The smaller the nanofiber diameter, the higher the tensile strength of the membrane. This trend aligns with the findings of previous studies on nanofiber membranes [15]. However, the relationship between nanofiber diameter and membrane tensile strength in this study was the opposite, as summarized in Table 2. The increase in tensile strength not only correlates with the nanofiber diameter but is also due to a potential increase in the density of cross-linking between nanofibers (Figure 3). An increase in the density of cross-linked fibers leads to the improvement of the membrane's tensile strength [16].

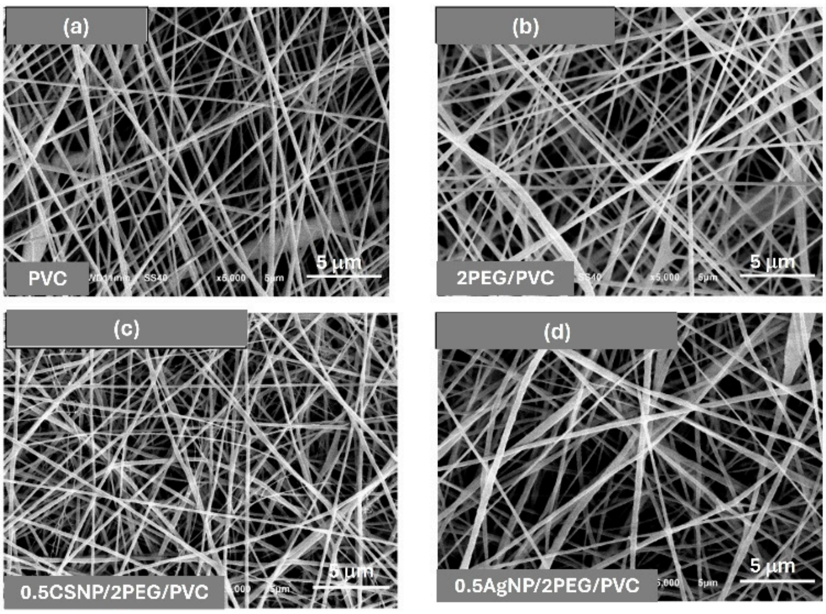

Figure 3. SEM images of the nanofiber membranes. (**a**) PVC, (**b**) 2PEG/PVC, (**c**) 0.5CSNPs/2PEG/PVC, and (**d**) 0.5AgNPs/2PEG/PVC.

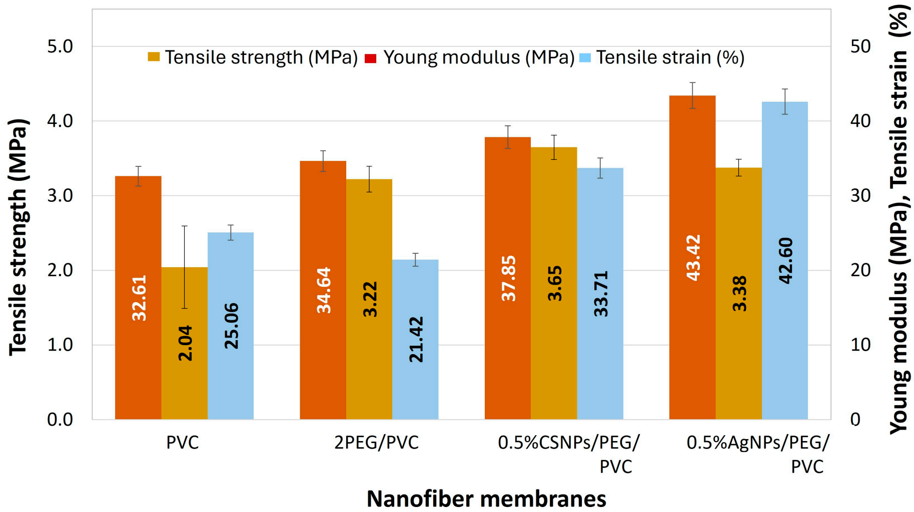

Figure 4. Tensile properties of the nanofiber membranes.

3.3. Water Filtration

Furthermore, for water filtration applications, only the nanofiber membranes 0.5CSNP/2PEG/PVC and 0.5AgNP/2PEG/PVC are used because they contain CS and Ag antibacterial agents. According to the number of bacteria recorded before and after water filtration and the filtration efficiency calculated by Equation (1) shown in Table 3, the bacterial test on groundwater indicated that the 0.5%AgNPs/PEG/PVC membrane worked better than the 0.5% CSNPs/PEG/PVC membrane in binding or killing Colitinja bacteria, while both types of membranes demonstrated a balanced ability to bind *E. coli* bacteria. However, the SEM results on the two membranes after the filtration test (Figure 5) showed a different morphology than before the filtration test (Figure 3). These results suggest the membranes worked well in binding bacteria during the filtration process as marked by arrows (Figure 5). However, the morphology of bound bacteria does not always show a clear image, as reported in the previous studies [17,18].

Table 3. Testing results for bacteria contained in the groundwater.

Nanofiber Membrane	Bacteria Testing (*E. coli* dan Colitinja, MPN/100 mL)			
	Before Filtration		After Filtration	
	E. coli	Colitinja	*E. coli*	Colitinja
PVC	≥1600	170	-	-
1PEG/PVC			-	-
2PEG/PVC			-	-
0.5CSNPs/2PEG/PVC			350 (η = ~78%)	70 (η = ~59%)
0.5AgNPs/2PEG/PVC			350 (η = ~78%)	< 1.8 (η = ~99%)

MPN: the m MPNMPN: the most probable number.

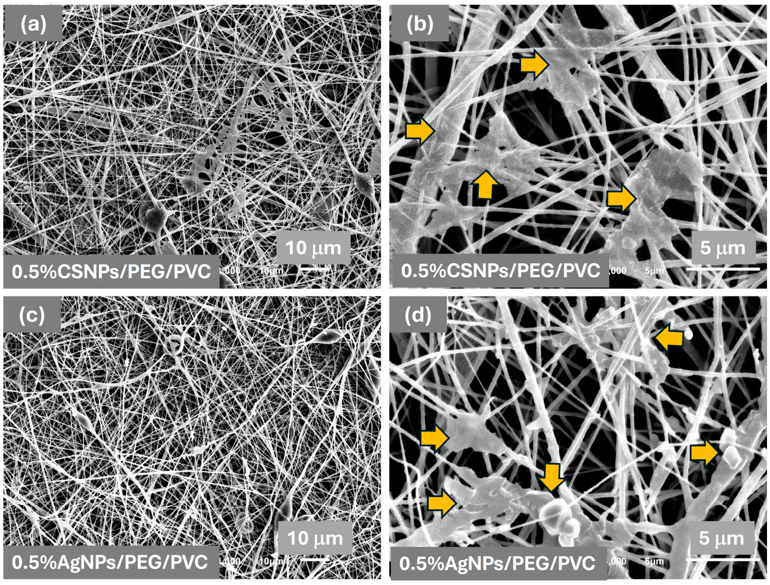

Figure 5. SEM images of 0.5CSNP/2PEG/PVC and 0.5AgNP/2PEG/PVC nanofiber membranes used after water filtration. The images of (**b**) and (**d**) are magnified versions of (**a**) and (**c**), respectively.

4. Conclusions

We have successfully fabricated, characterized, and applied PVC-based nanofiber membranes (PEG/PVC, CSNPs/PEG/PVC, and AgNPs/PEG/PVC) as water filters. The addition of PEG to PVC (2PEG/PVC) significantly decreased the water contact angle and increased nanofiber diameter, tensile strength, and Young's modulus while decreasing the tensile strain. Meanwhile, adding CSNPs and AgNPs to the 2PEG/PVC membrane raised all these parameters. The use of 0.5CSNPs/2PEG/PVC and 0.5CAgNPs/2PEG/PVC membranes for filtering groundwater was very effective at inhibiting the growth of *E. coli* and Colitinja bacteria. Additionally, the 0.5%AgNPs/PEG/PVC membrane is much more effective in binding and killing Colitinja bacteria than the 0.5%CSNPs/PEG/PVC membrane, but the binding ability of *E. coli* bacteria on both types of membranes is balanced. Therefore, the addition of CSNPs and AgNPs to nanofiber membranes holds the potential for further development as water filter materials.

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